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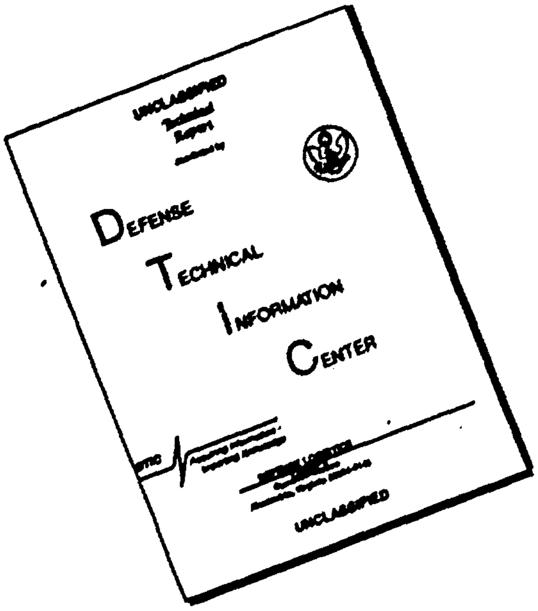

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U. S. Army

Chemical Warfare Laboratories

Technical Report

CWLR

Cold Gas Atomization of Low Volatility Liquids

by

Malcolm G. Gordon

January 1960

ARMY CHEMICAL CENTER, MD

January 1960 CWLR 2339

COLD GAS ATOMIZATION OF LOW VOLATILITY LIQUIDS

by

Malcolm G. Gordon

Weapons Research Division

Recommending Approval

Carl M Herget

CARL M. HERGET, Ph.D.
Acting Director of Research

Approved:

S. D. Silver

S. D. SILVER
Deputy Commander for Scientific Activities

U. S. ARMY
Chemical Corps Research and Development Command
CHEMICAL WARFARE LABORATORIES
Army Chemical Center, Maryland

FOREWORD

This work was conducted under Subproject 4-04-15-029-02, Dissemination and Exploitation of CW Agents (U). The experimental data are recorded in notebook 4927. The work was started in January 1956 and completed in March 1957. This report was submitted for publication in May 1959.

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DIGEST

The object of the work described in this report was to study the factors affecting the atomization of nonvolatile liquids with cold gas (compressed air).

Bis(2-ethylhexyl)hydrogen phosphite was atomized by compressed air using a device similar in design to a multicompartment thermal generator and equipped with various nozzle configurations. Several different sampling techniques were used as a means of characterizing the aerosol produced. The data obtained were compared to the Nukiyama-Tanasawa equation characterizing the process, and the application of the cold gas technique to a practical munition device was considered.

As a result of the investigation conducted, the following conclusions were drawn:

(1) The aerosol produced by the compressed-air-operated laboratory device can be characterized for most of the nozzle designs considered by the Nukiyama-Tanasawa relationship.

(2) The parameter most significantly affecting the atomization process for a given liquid is the volume ratio of liquid to gas.

(3) The four sampling techniques examined presented varying degrees of correlation to each other and to the value predicted by the Nukiyama-Tanasawa relationship. The swinging-arm technique appeared to be the most suitable on the basis of accuracy and ease of operation.

(4) The cold gas atomization technique considered did not appear to be practicable for incorporation into a chemical munition because of low liquid capacity; this technique, however, provides a means of producing, without chemical decomposition, an aerosol having the desired characteristics.

CONTENTS

COLD GAS ATOMIZATION OF LOW VOLATILITY LIQUIDS

I. INTRODUCTION.

A. Object.

The object of the work described in this report was to study the factors affecting the atomization of nonvolatile liquids with cold gas (compressed air).

B. Background.

In the operation of a thermal generator, a liquid is inserted into the hot gas stream produced by a burning pyrotechnic. The hot gas is directed at high velocity through a nozzle that may be of either venturi or straight-tube design. The liquid injected into the gas stream in the nozzle is thus both atomized and vaporized. The vapor thus produced subsequently condenses in the atmosphere to produce an aerosol.

Although some performance data of the venturi-type thermal generator in disseminating a nonvolatile CW agent had been obtained, no correlation was established between the operating conditions of the device and the aerosol produced. Through minor design changes in the unit, the aerosols produced have ranged from an aerosol consisting primarily of particles of from 10μ to 200μ in diameter to an aerosol having from 50% to 70%, by weight, of particles less than 10μ in diameter.[1]

During previous experimentation with this test device, measurement of the variables believed important in producing the desired aerosol was quite difficult. The interdependence of such variables as gas-flow rate, fuel-block burning pressure, and temperature, for example, did not permit the desired control of performance. Moreover, variations in the aerosol produced could not be established because of either device-operation or aerosol-assessment techniques.

Accordingly, the work reported herein was initiated (1) to determine the variables that significantly affect atomization without the influence of high gas temperatures, and (2) to evaluate the various aerosol-sampling techniques as applied to these aerosol-producing devices. A series of experiments was conducted with a cold-gas (compressed-air) atomizing device using the basic hardware design of a thermal generator

equipped with various nozzle configurations (figures 1 and 2, appendix).

The field of compressed-air liquid atomization is one in which considerable effort has been expended. Many investigators have examined the mechanism of aerosol formation, sampling, and evaluation, and a broad review of the subject may be found in the literature.[2] It is generally agreed, however, that some of the more important factors governing atomization include the ratio of liquid to gas, relative velocity of liquid to gas, and the physical properties of the liquid and gas.

Review of the literature pertinent to atomization indicated the general applicability of an empirical equation derived by Nukiyama and Tanasawa:[3]

$$D_o = \frac{585\sqrt{\sigma}}{v\sqrt{p}} + 597\left(\frac{u}{\sqrt{\sigma p}}\right)^{0.45}\left(\frac{1000\,Q_L}{Q_G}\right)^{1.5} \qquad (1)$$

where

D_o	=	Sauter mean diameter, microns
v	=	velocity difference between liquid and gas, m/sec
Q_L	=	volume of liquid per unit time, ml/sec
Q_G	=	volume of gas per unit time, ml/sec
p	=	density of liquid, gm/ml
u	=	viscosity of liquid, poise
σ	=	surface tension of liquid, dynes/sq cm

Generally verified by these and other investigators was a limiting ratio of Q_L/Q_G, the validity of the equation for velocities of subsonic and sonic gases, and the independence of nozzle configurations.[1,2,3,4,5] In determining the atomization characteristics of a nozzle design somewhat different from those previously examined, it was believed that equation (1) could still be utilized. Therefore, the measurement and the theoretical significance of the equation parameters were necessary.

Inherent in determining the parameters describing an aerosol distribution, for example, Sauter mean diameter (D_o) or mass median diameter (MMD), are the errors associated with sampling and the statistical error of any slide particle analysis. The technique of simply passing a microscope slide through the atomized jet issuing from a nozzle has been rather successful in sampling the aerosol produced. Because of decreasing impaction efficiency with decreasing particle size, however, any calculation of D_o using this technique would be fallaciously high if a significant quantity of the mass is less than 10μ in size. Moreover, the problems of obtaining a statistically valid particle-size distribution from slides are recognized, and only a sufficiently large sample will adequately characterize the larger or tail end of a distribution that significantly influences any value of D_o or MMD. As an aid in reducing the aforementioned errors, other techniques of aerosol sampling were used to supplement the single waved slide. These techniques used floor slides and air-sampling filters after specific periods of stirred settling.

As a convenient procedure in calculating experimental values of D_o from equation (1), a graphical solution requiring two charts was prepared. Equation (1) reduces to:

$$D_o = b + (c)(a)^{1.5} \qquad (2)$$

where

$$b = \frac{585\sqrt{\sigma}}{v\sqrt{P}} \qquad (2A)$$

$$c = 597\left(\frac{u}{\sqrt{\sigma P}}\right)^{0.45} \qquad (2B)$$

$$a = 1000\, Q_L/Q_G \qquad (2C)$$

Rearranging equation (2) and putting it into logarithmic form:

$$\log (D_o - b) = \log c + 1.5 \log a \qquad (2D)$$

By substituting the physical constants of the liquid, bis (2-ethylhexyl)hydrogen phosphite, equation (2A) can be rearranged, and b versus v can be plotted (figure 3, appendix).

Similarly, equation (2D) can be plotted as (D_0-b) versus (1000 Q_L/Q_G) (figure 4, appendix). Thus, knowing v (and b) and the ratio (1000 Q_L/Q_G), D_o can be interpolated directly. The above relation was calculated for liquid temperatures of 20°C representing temperate conditions, and for 7°C, representing the general temperature level during winter experimentation.

II. EXPERIMENTATION.

A. Test Chamber.

The aerosol-dissemination chamber used in this program measures 16 by 16 by 12 feet and has a volume of 87 cu m. The chamber is equipped with compressed-air tanks and has facilities for the measurement of pressure, flow rate, and temperature.

The chamber floor is marked in a symmetrical, circular, floor-grid pattern for positioning the microscope slides utilized in aerosol evaluation (figure 5, appendix). Various geometric patterns consisting of a smaller number of slide positions were derived from this basic pattern. Nine positions were marked on the chamber walls. These positions were in the center of nine equal wall areas on the lower half of each wall.

B. Materials and Equipment.

1. Aerosol-Sampling and Measuring Devices.

a. Air-Sampling Devices.

Filter equipment consisted of two filters as a pair at the 2-, 4-, and 6-foot levels on a pole. Generally, three such sampling poles were utilized. A Cenco Cat. No. 93970 vacuum pump was used for the six samplers on each pole. Critical orifices controlled the air flow through each filter. One layer of 50% Vitron 112 and 50% Vitron 106, manufactured by Glass Fibers, Inc., was used for sampling. In some experiments, liquid bubblers for vapor sampling were used in series behind the filters.

b. Impactmeter.

Some success has been attained by other investigators in sampling the aerosol produced by an atomizing device by simply exposing a microscope slide in the issuing jet.[1,2,3,4,5] Based on this principle, a

device called an Impactmeter was designed. It rotated slides under an open 1-inch-wide slot positioned at the approximate center of the atomizing jet (figure 6, appendix). The slides were rotated at approximately 3 ft/sec under the slot.

Based on the same principle was a swinging arm, which moved laterally across the aerosol issuing from the atomizing nozzle. Three microscope slides were mounted on the arm so that the center and points midway between the center and circumference of the aerosol stream were crossed. The velocity of the arm was approximately 3 ft/sec.

c. Floor Slides.

Standard 3-inch by 1-inch microscope slides were used for particle analysis and material balances. The particles deposited on the floor slides were counted and measured by a device described in a previous report.[6]

2. Atomizing Device and Nozzles.

The device used for the compressed-air atomization is illustrated in figure 1, appendix. The various nozzle designs are shown in figure 2, appendix.

3. Instrumentation.

Pressure measurements were accomplished by a Sanborn Model 127 recorder and various commercial gauges. Compressed-air-flow rates were determined by the use of a 1-inch, sharp-edged orifice in a 2-inch standard pipe with pressure taps connected to a U-tube manometer. Liquid-flow rates were determined from data of the duration and quantity of feed.

4. V-Agent Simulant.

The liquid used in all experimentation was bis(2-ethylhexyl) hydrogen phosphite.

C. Procedure.

1. Compressed-Air Atomization.

A device similar in design to a multicompartment thermal generator and equipped with nozzles of various configurations was used in the compressed-air atomization of the liquid. Air-flow rates were determined

prior to experimentation as a function of chamber pressure and liquid-feed pressure. Plotting these data permitted interpolation of air-flow rates for any operational condition (figure 7, appendix).

The atomizing device was suspended from the ceiling of the chamber, and the aerosol produced was directed vertically downward. During aerosol production, the Impactmeter and arm-sampling devices were utilized both in conjunction with the floor slides and separately in duplicate experiments. Upon completion of dissemination the aerosol was subject to stirred settling for a period of 30 minutes. Filter samples were obtained during the last 3 minutes of this period. Based on spherical-particle-settling laws, air-sampling data at this particular time indicated that the quantity of material was generally less than 10μ in size. Upon removal from the chamber, the floor slides were examined for particle-size measurements and later chemically analyzed for material-balance calculations. The material-balance calculations were supplemented by the air-filter data and additional slides were placed on the chamber walls.

All glass slides used for particle-size analysis were cleaned and processed by May's method[7] prior to use. Droplet-spread factors (the ratio of the diameter of the droplet as a sphere in the air to the diameter of the same droplet while on the glass slide) were also determined by May's method. These experimental data compared within 5% of data obtained by a completely different technique.[8]

2. Calculations.

a. Particle Size.

As noted in figure 5, appendix, there were 45 sampling slide positions. Each slide was examined, and a minimum number of 500 particles were counted and measured. The device used in particle counting and in the microscope-projection magnification was adjusted to the particles could be separated into 17 intervals according to size. The projected magnification was 300 power, which permitted measurement of particles from 4μ to 175μ in intervals of approximately 10μ each.

The same total area on the slides was examined, and in those instances where a larger slide area was necessary to maintain the minimum of 500 particles, the data were adjusted to an equal area basis. In figure 5, appendix, the solid circular lines depicting annuli were considered as being represented by the slides within them. For example, the eight slides of

ring 1 represent the annular area between circles having radii extending to the midpoint between ring 0 and ring 1 and between ring 1 and ring 2. All the slide particle-size data of a ring were multiplied by a factor corresponding to the ratio of annuli areas. The factors for the 3- by 1-inch slides are shown in table 1.

TABLE 1

AREA FACTOR FOR PARTICLE-SIZE CALCULATION

Ring	Annulus area	Area per slide	Area factor
	sq in.		
0	113	113	1.0
1	905	113	1.0
2	3054	382	3.38
3	7240	905	8.0
4	10,860	1360	12.0
5	11,270	1410	12.5
6	3420	356	7.6

Upon completion of adjustment for the slide area examined and floor area represented, the numbers of particles in each size interval were totaled. The totals and the corresponding particle-size-interval midpoint were the basis for calculation of D_o from the general equation:

$$D_o = \frac{\Sigma nd^3}{\Sigma nd^2} \quad (3)$$

where

n = the number of particles in a size interval

d = the midpoint diameter of a size interval

The same general type of calculation was used when other geometric patterns of floor slides were utilized. In determining D_0 from the Impactmeter and arm-sampling slides, the counting data obtained were totaled, and D_0 was calculated directly.

b. Material Balance.

Upon completion of slide particle analysis, the floor and wall slides were chemically analyzed for liquid agent. The total material on all slides of a grid ring was divided by the total area of the same slides yielding a concentration per square inch. This value of floor concentration was representative of the annulus area corresponding to the ring. The quantity of agent in each annulus area was summed to give the total quantity on the floor.

The same general type of calculation was performed for the slides positioned on the chamber walls. The liquid airborne at 30 minutes was calculated directly from filter-sample analysis and sampling flow-rate data. Agent recovery was the percentage value of the liquid recovered, divided by the total liquid disseminated.

D. Results.

1. Aerosol Particle-Size Analysis.

The aerosol data obtained from the compressed-air atomizing device are plotted in figures 8, 9, and 10, appendix, as is the value of D_0 as predicted by the Nukiyama-Tanasawa equation from the operating conditions. Also shown for purposes of comparison in figure 8, appendix, are the values of D_0 as computed from the Impactmeter, arm, and the 45- and 16-floor slide patterns. The generalized aerosol distributions, as a function of Q_L in ml/sec taken from the data in figure 8, appendix, are plotted in figure 11, appendix. A logarithmic-normal distribution was assumed for the aerosol distributions for purposes of presentation. The exact type of distribution was not determined, but the experimental data were closely approximated by the logarithmic-normal curve. The aerosol data illustrated in figures 9 and 10, appendix, were also closely approximated by a logarithmic-normal distribution.

2. Material Balance.

The experimental data obtained in performing the chamber material balances are shown in table 2.

TABLE 2

AEROSOL CHAMBER MATERIAL BALANCES

Code	Total liquid disseminated	Liquid airborne	Liquid on walls	Liquid in bubblers	Liquid on floor				Total liquid recovered	
					45 slides		16 slides		45 slides	16 slides
	gm				gm	%	gm	%	%	
11/21	63	3.0	7.0	0	46	73	--	--	89	--
12/3	63	5.0	6.0	0	47	75	50	80	92	97
12/9	34	4.4	4.4	0	19	56	23	68	82	94
12/17	62	4.4	7.0	–	42	68	54	89	86	100
12/19	65	1.0	7.0	–	37	57	45	69	70	81

III. APPLICATION OF ATOMIZATION EXPERIMENTATION.

Based on the experimental data, it was of interest to determine the basic design of a compressed-air munition device capable of producing specific types of aerosols. The general procedure followed was to determine the Q_G/Q_L ratio required to produce a given aerosol and then to calculate the necessary relative volumes of liquid and gas. For example, in figure 8, appendix, a ratio of $Q_G/Q_L = 4000$ was required to produce an aerosol with a D_o value of 20μ. From figure 4, appendix, an aerosol distribution with an MMD value (cumulative mass of 50%) of 20μ was interpolated. This aerosol distribution, which has 95% of its mass in particles smaller than 40μ, was produced at a flow rate of 5-ml/sec with the experimental system. D_o and MMD were assumed to be approximately equal in this relatively narrow particle-size distribution. If Q_L were 5 ml/sec then Q_G would have to be (4000 x 5) or 20,000 ml/sec at ambient conditions.

A munition device with a total available volume of 1000 ml was assumed, and the relative volumes of liquid and gas (at various holding pressures) and munition wall thickness (calculated by hoop tension) were determined. Similar calculations were performed in analyzing the system with nozzles similar to those in figure 2, appendix, items a and b, which could produce an aerosol with a D_o of 40μ (Q_G/Q_L = 2000) and a D_o of 90μ (Q_G/Q_L = 1000). In these latter two examples D_o was also assumed to be equivalent to MMD, although, more rigorously, MMD should be calculated from the particle-size-distribution data that yielded the value of D_o. Generally, the experimental data indicated that MMD/D_o was from 1.0 to 1.2 in the D_o range of 15μ to 200μ. The results of these calculations are shown in table 3 and indicate the impracticability of the munition system for the production of aerosols because of the low liquid capacity. The use of a gas other than compressed air, for example, helium or carbon dioxide, or the use of nozzles of other designs may prove more practicable.

IV. DISCUSSION.

The characteristics of the aerosols produced by the atomizing device, for most of the nozzles considered, agree well with the D_o values predicted by the Nukiyama-Tanasawa equation. The performance data of all nozzles indicated a limiting ratio of approximately $Q_G/1000\ Q_L$ = 5. Larger values of the ratio did not produce particles smaller than 13μ to 15μ, a fact that confirms earlier data of other investigators.[3] The performance of the small venturi and coiled-tube nozzles, however, yields values of D_o that are consistently lower than expected at $Q_G/1000\ Q_L$ ratios less than 5. The data

TABLE 3

LOAD AND PHYSICAL CHARACTERISTICS OF AN ATOMIZING MUNITION DEVICE

Air pressure	Approximate wall thickness of device	Air volume at STP	Air volume in device	Liquid capacity
psig	inch	10^3 ml	ml	ml
		$Q_G/Q_L = 4000$, $D_o = 20\mu$		
2,000	0.06	130	968	32
5,000	0.15	310	923	77
10,000	0.30	570	857	143
		$Q_G/Q_L = 2000$, $D_o = 40\mu$		
2,000	0.06	125	937	63
5,000	0.15	290	857	143
10,000	0.30	500	750	250
		$Q_G/Q_L = 1000$, $D_o = 90\mu$		
2,000	0.06	120	882	118
5,000	0.15	250	750	250
10,000	0.30	400	600	400

of both nozzles are well beyond the estimated experimental error. Since the coiled-tube-nozzle design is rather unconventional, abnormal results might have been expected but data of the small venturi nozzle are contrary to earlier findings.[5] Although no firm explanation can be proposed, it is believed that the flow conditions within the nozzle, i.e., velocity changes, and the magnitude and position of standing pressure waves, may have been contributing factors.

The variation of some of the experimental points illustrated in figures 8, 9, and 10, appendix, is partially the result of experimental error. In the region of $Q_G/1000\ Q_L = 1$, a very slight change in the ratio causes a large variation in D_o. The experimental precision in the measurement of Q_G and Q_L was such that closer agreement could not be attained, and, therefore, the variation could be expected. It will be noticed that the points in this region also fall below the Nukiyama-Tanasawa curve rather than on both sides. It is believed that this condition is caused by incomplete measurement of the larger particles existing in the aerosol distributions. If a more representative number of the larger particles had been observed and counted, the D_o values shown would have been greater.

The same general type of statistical error probably existed in the range of $Q_G/1000\ Q_L = 5$. In this region of relatively small particles, $<30\mu$, insufficient very small particles actually impacted on the slides of the Impactmeter, thus influencing the experimental D_o towards a value greater than the Nukiyama-Tanasawa predicted value. If these data were to be adjusted on the basis of the quantity of liquid airborne as shown in table 2, the values of D_o would be decreased as much as 15%. Also, this series of experiments was performed in colder weather and, as seen in figure 3, appendix, the decrease from summer to winter temperatures increased the theoretical value of D_o approximately 15%.

Generally, the data presented in figure 8, appendix, indicate reasonable sampling validity of any of the four techniques used. Floor-slide sampling would be expected to yield the most representative data. The 45- and 16-floor slide patterns indicated good greement between both the pattern and the Nukiyama-Tanasawa predicted value. Impactmeter data appeared more variable, whereas, the swinging-arm-slide data appeared to be more consistent. Possibly, the manner of sampling made it feasible to take a more representative aerosol-particle sample with the swinging-arm technique than with the Impactmeter.

It is obvious from table 2 that sampling by the floor-slide techniques accounted for slightly more than half the total liquid disseminated.

From the apparent correlation of the experimental data to the theoretical information shown in figures 8, 9, and 10, appendix, it is believed that the experimental values obtained were reasonably representative of the entire aerosol produced.

V. CONCLUSIONS.

As a result of the investigation conducted, the following conclusions were drawn:

1. The aerosol produced by the compressed-air-operated laboratory device can be characterized for most of the nozzle designs considered by the Nukiyama-Tanasawa relationship.

2. The parameter most significantly affecting the atomization process for a given liquid is the volume ratio of liquid to gas.

3. The four sampling techniques examined presented varying degrees of correlation to each other and to the value predicted by the Nukiyama-Tanasawa relation. The swinging-arm technique appeared to be most suitable on the basis of accuracy and ease of operation.

4. The cold gas atomization technique considered did not appear to be practicable for incorporation into a chemical munition because of low liquid capacity; this technique, however, provides a means of producing, without chemical decomposition, an aerosol having the desired characteristics.

VI. LITERATURE CITED.

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APPENDIX

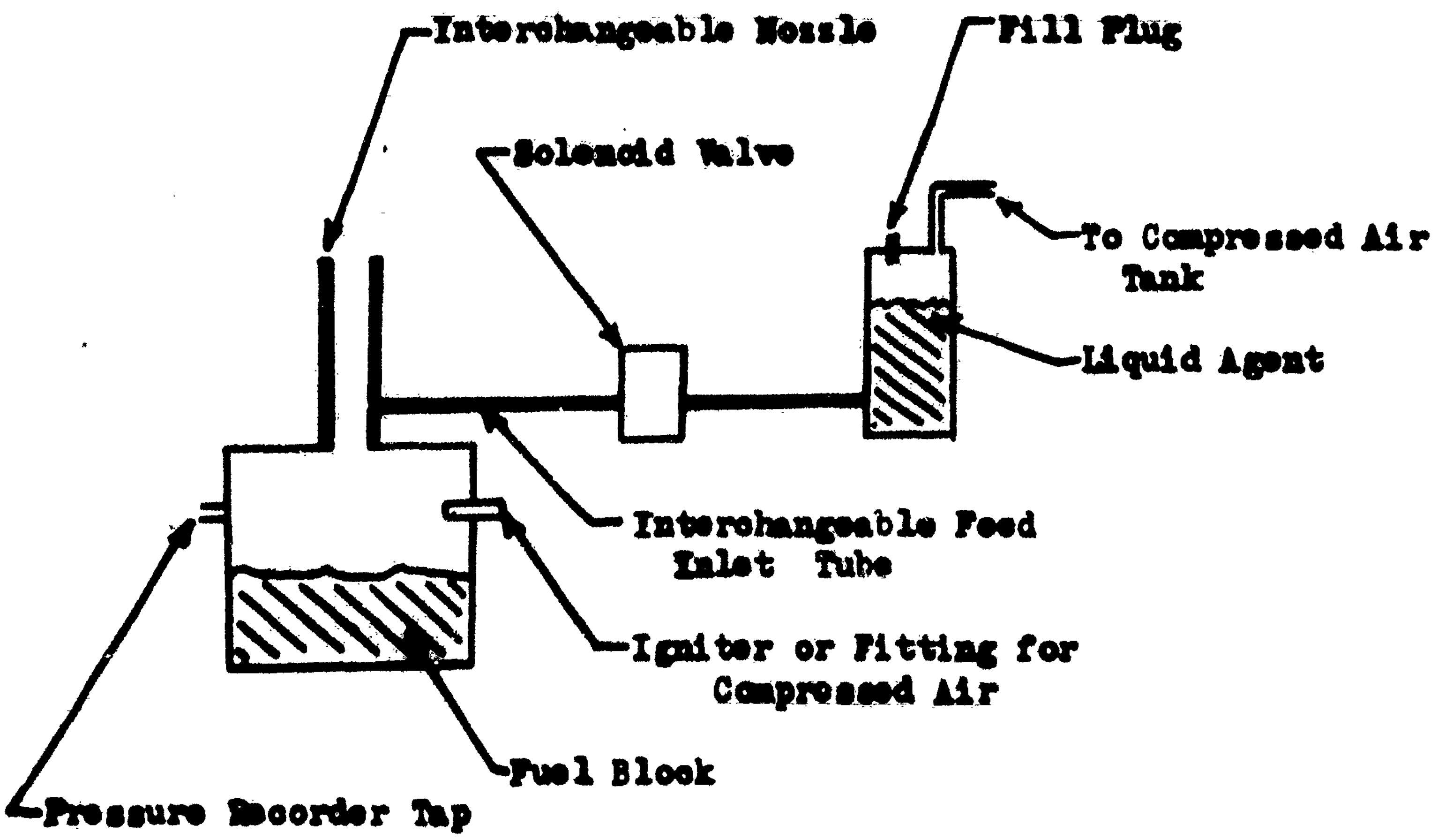
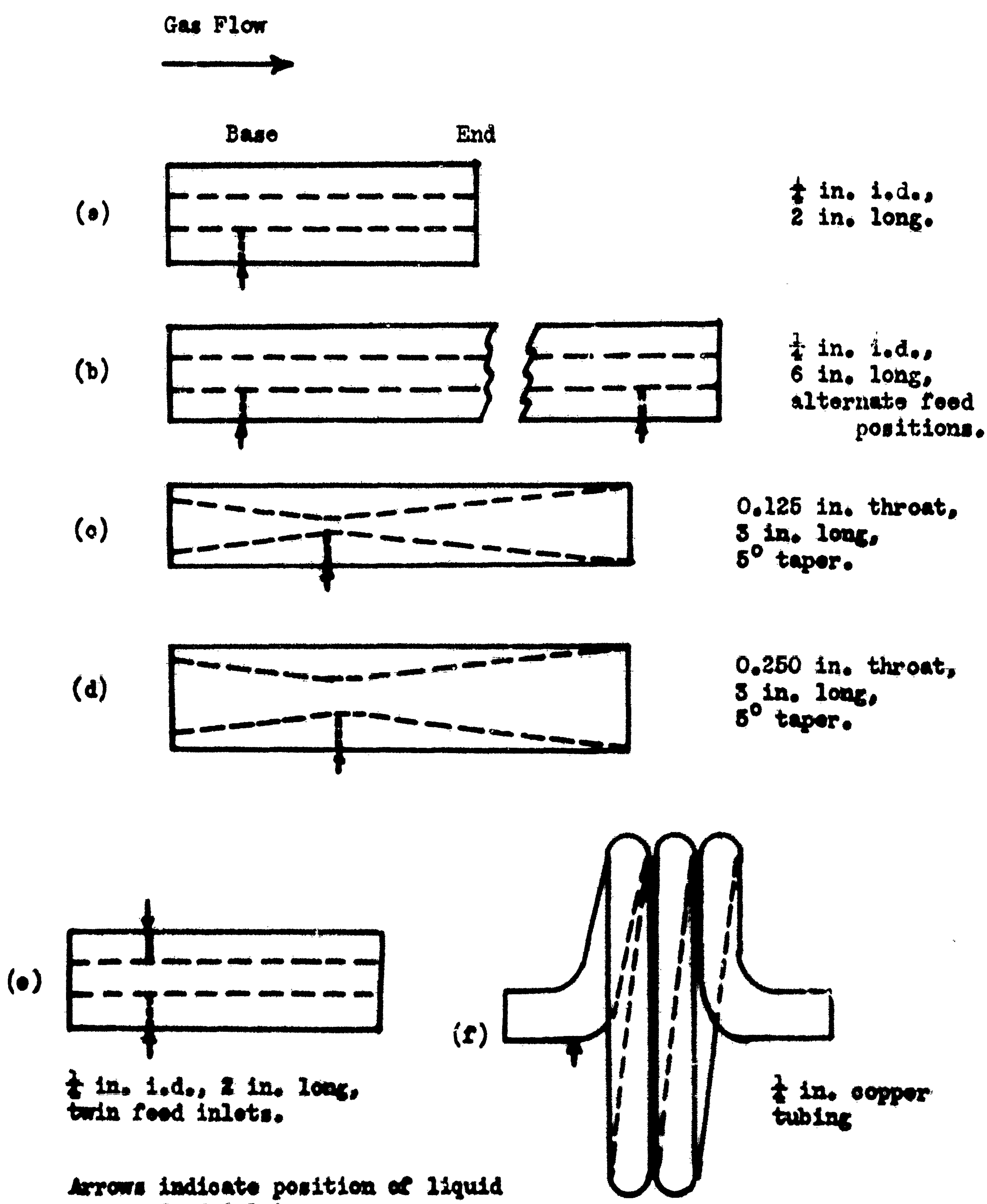


FIGURE 1

LABORATORY THERMAL GENERATOR, E-37



Arrows indicate position of liquid feed inlet.
Drawings are to scale.

FIGURE 2

NOZZLE DESIGNS

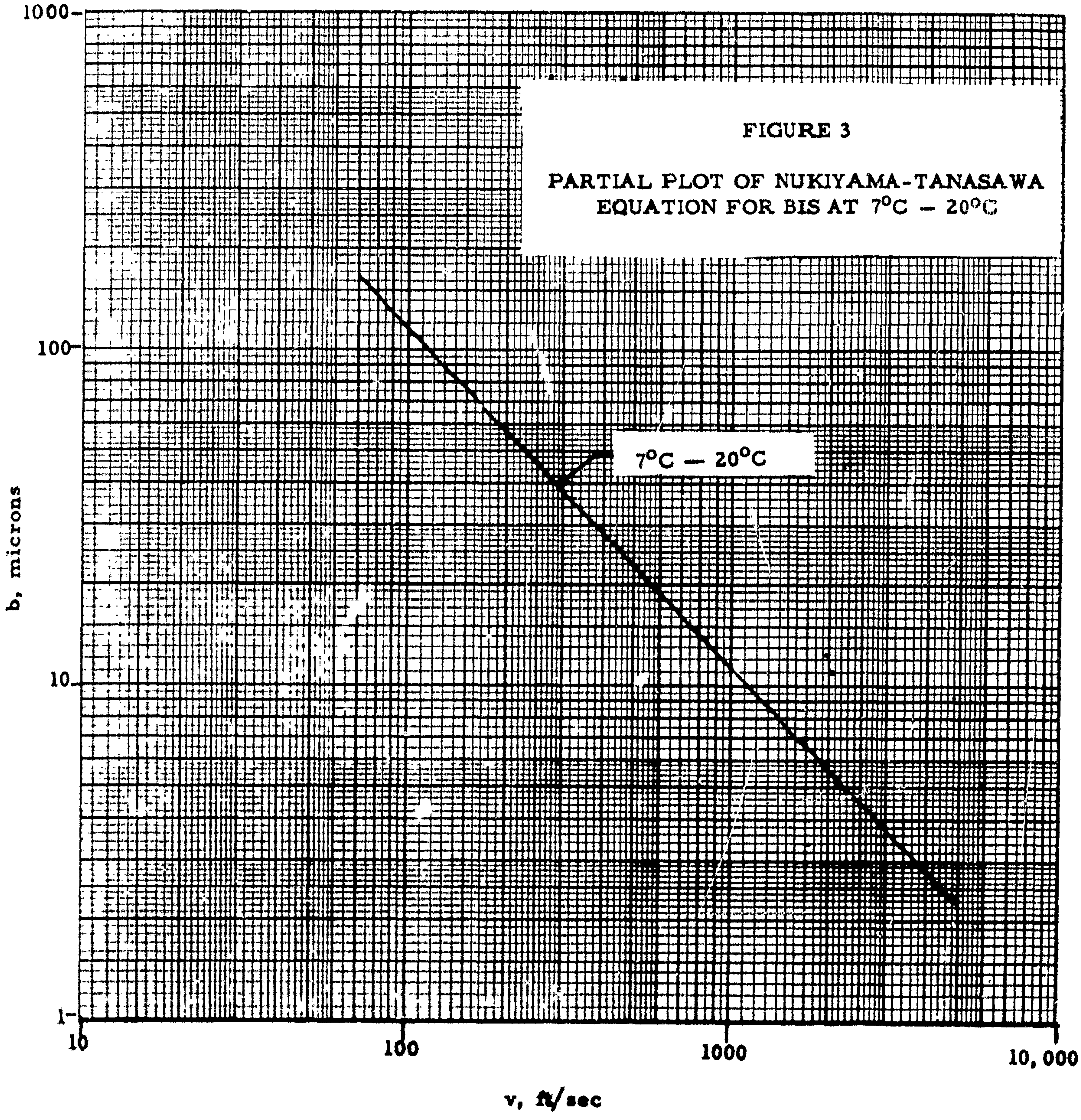


FIGURE 3
PARTIAL PLOT OF NUKIYAMA-TANASAWA EQUATION FOR BIS AT 7°C – 20°C
7°C – 20°C
1000
100
10
1
b, microns
10
100
1000
10,000
v, ft/sec

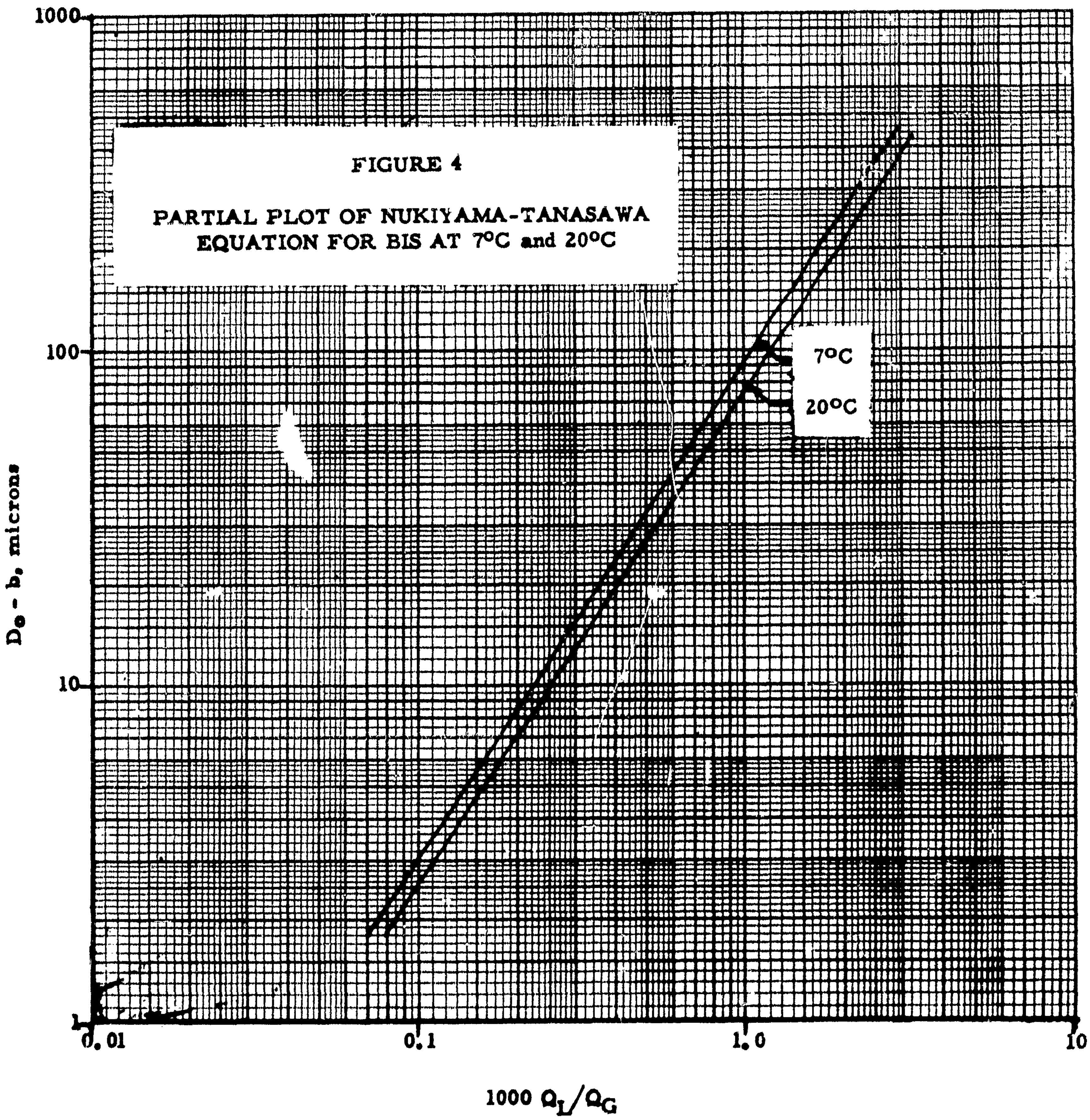


FIGURE 4
PARTIAL PLOT OF NUKIYAMA-TANASAWA EQUATION FOR BIS AT 7°C and 20°C
7°C
20°C
1000
100
10
1
0.01
0.1
1.0
10
D_o - b, microns
1000 Q_L/Q_G

Dimensions:

a = 12-in. radius
b = 24-in. radius
c = 48-in. radius
d = 72-in. radius
e = 96-in. radius
f = 120-in. radius

NOTE: Blocks indicate 45-slide pattern. Solid blocks indicate 16-slide pattern.

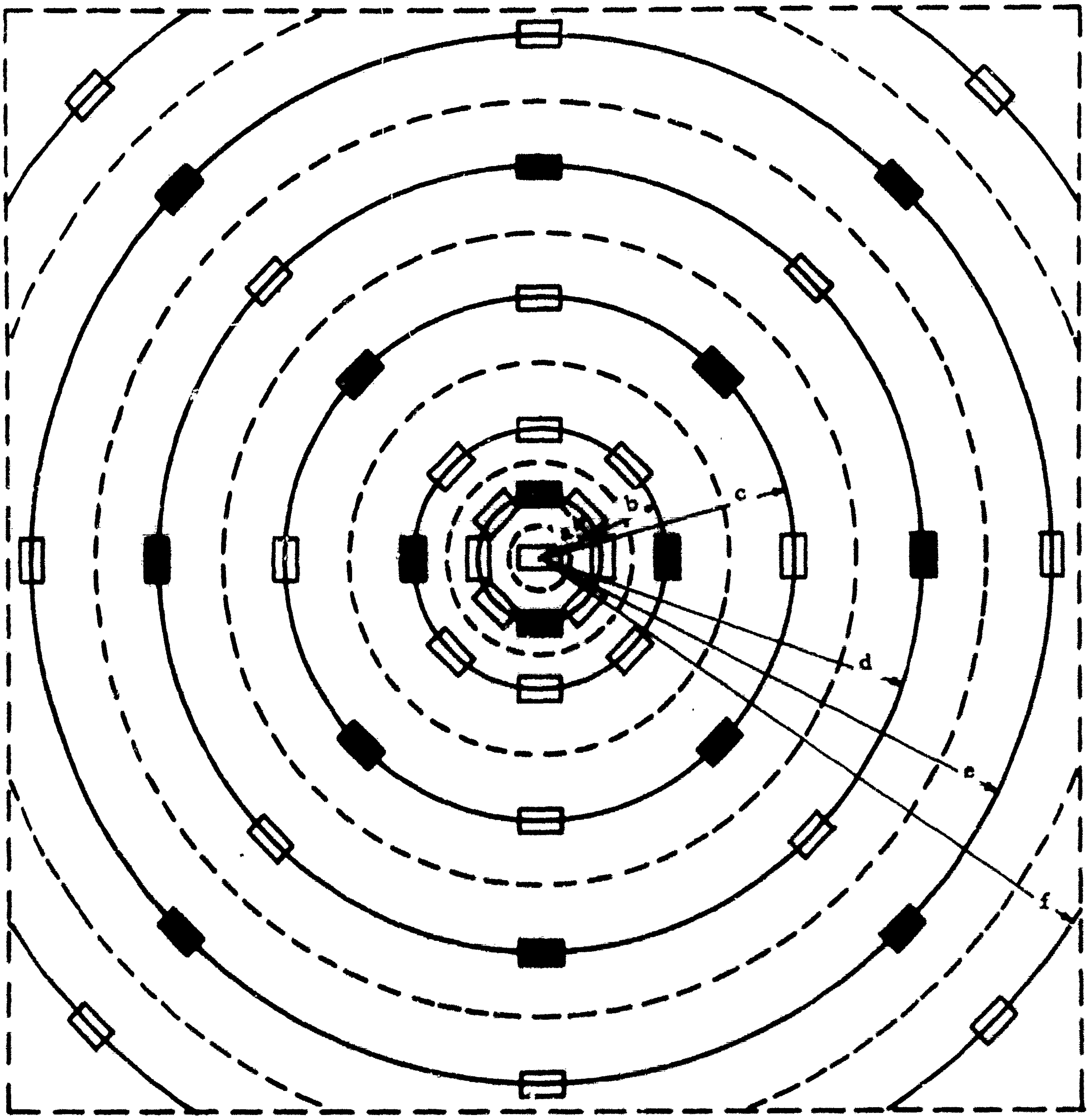


FIGURE 5

CHAMBER FLOOR-SLIDE GRID PATTERN

FIGURE 6

IMPACTMETER SHOWING ROTATING SLIDES AND COVER SLOT

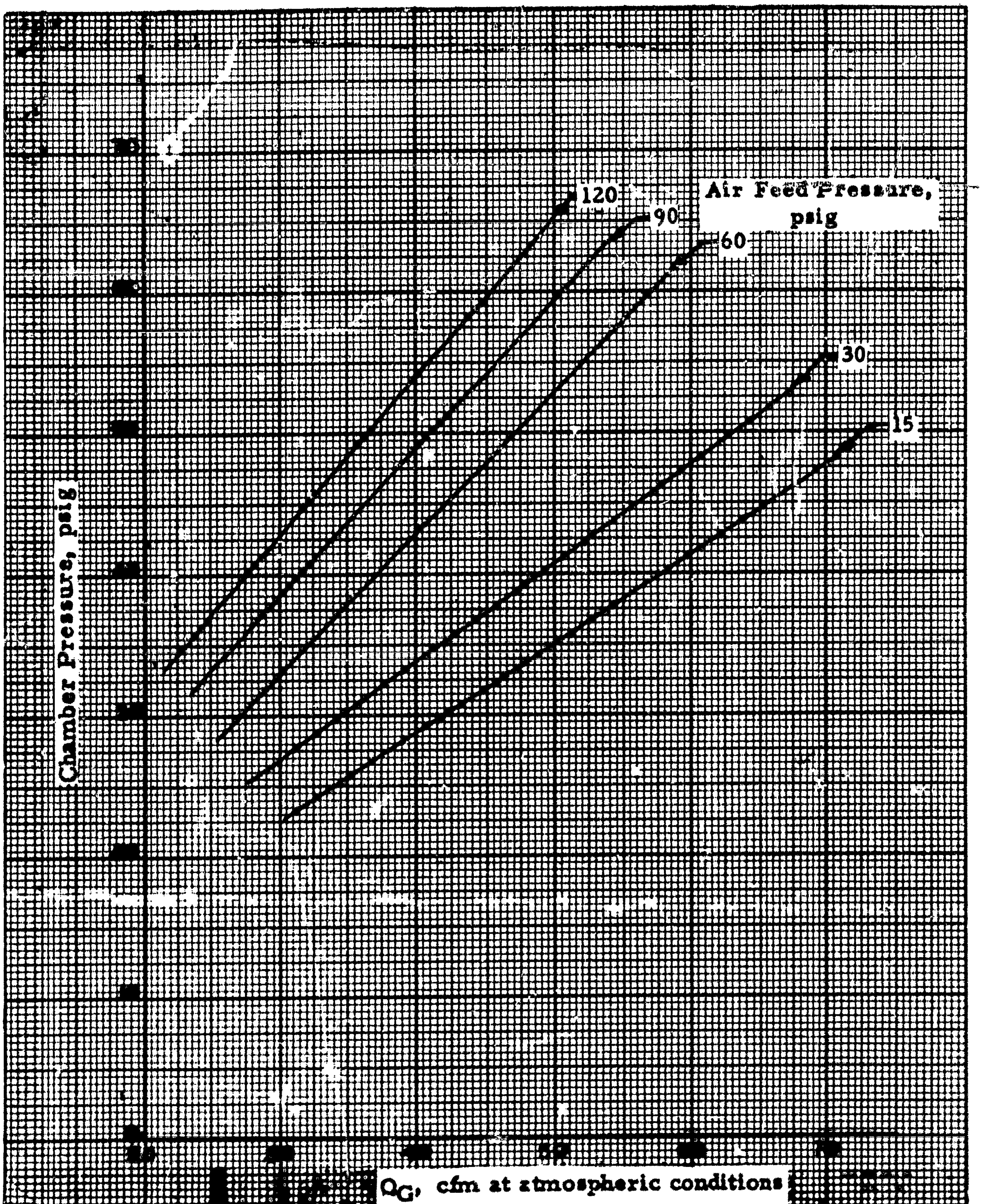


FIGURE 7

Q_G AS CFM AT ATMOSPHERIC CONDITIONS AS A FUNCTION OF CHAMBER AND AIR-FEED PRESSURES

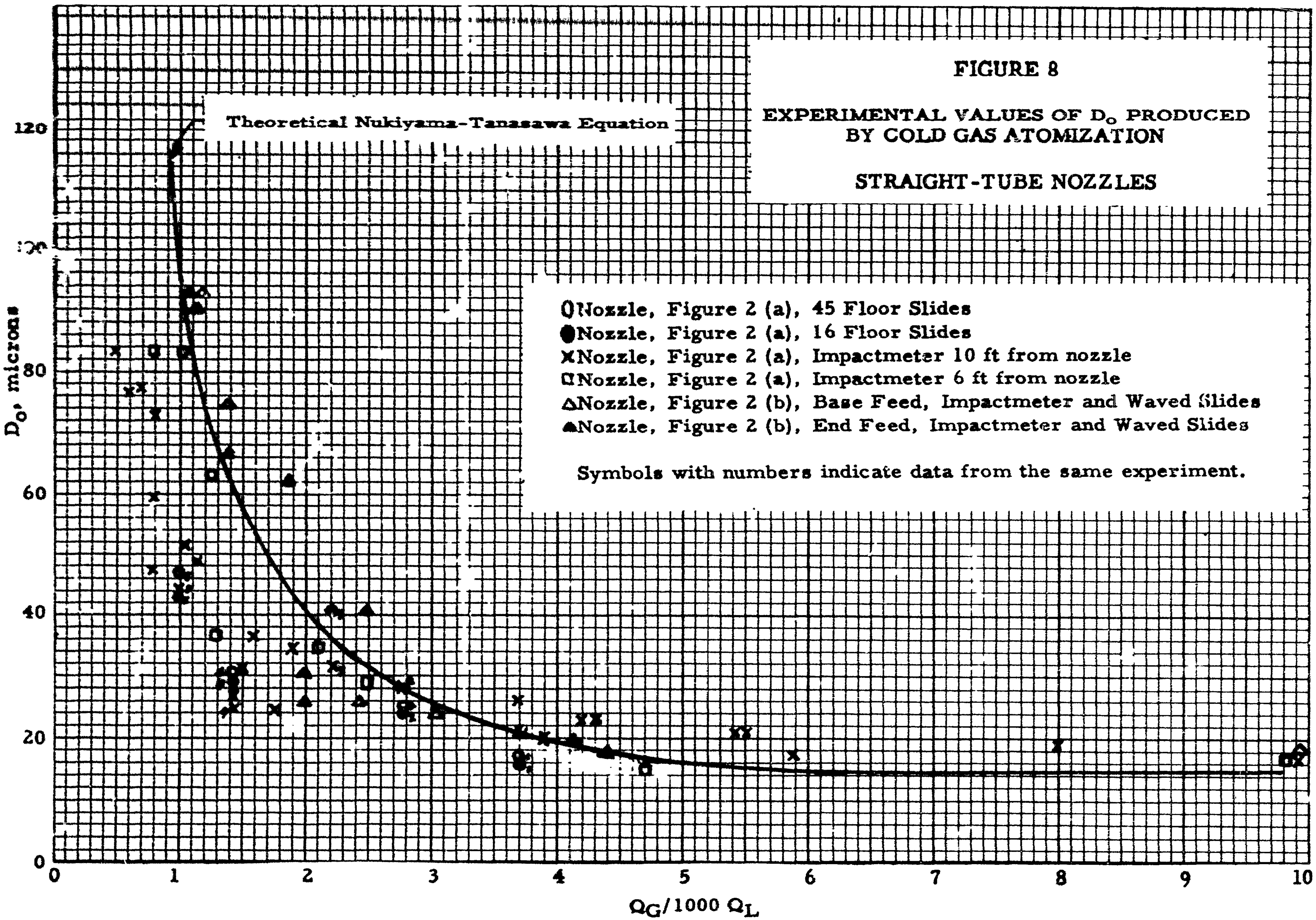


FIGURE 8
EXPERIMENTAL VALUES OF D_o PRODUCED BY COLD GAS ATOMIZATION
STRAIGHT-TUBE NOZZLES
Theoretical Nukiyama-Tanasawa Equation
○Nozzle, Figure 2 (a), 45 Floor Slides
●Nozzle, Figure 2 (a), 16 Floor Slides
×Nozzle, Figure 2 (a), Impactmeter 10 ft from nozzle
□Nozzle, Figure 2 (a), Impactmeter 6 ft from nozzle
△Nozzle, Figure 2 (b), Base Feed, Impactmeter and Waved Slides
▲Nozzle, Figure 2 (b), End Feed, Impactmeter and Waved Slides
Symbols with numbers indicate data from the same experiment.
D_o, microns
120
80
60
40
20
0
0
1
2
3
4
5
6
7
8
9
10
$Q_G/1000\ Q_L$

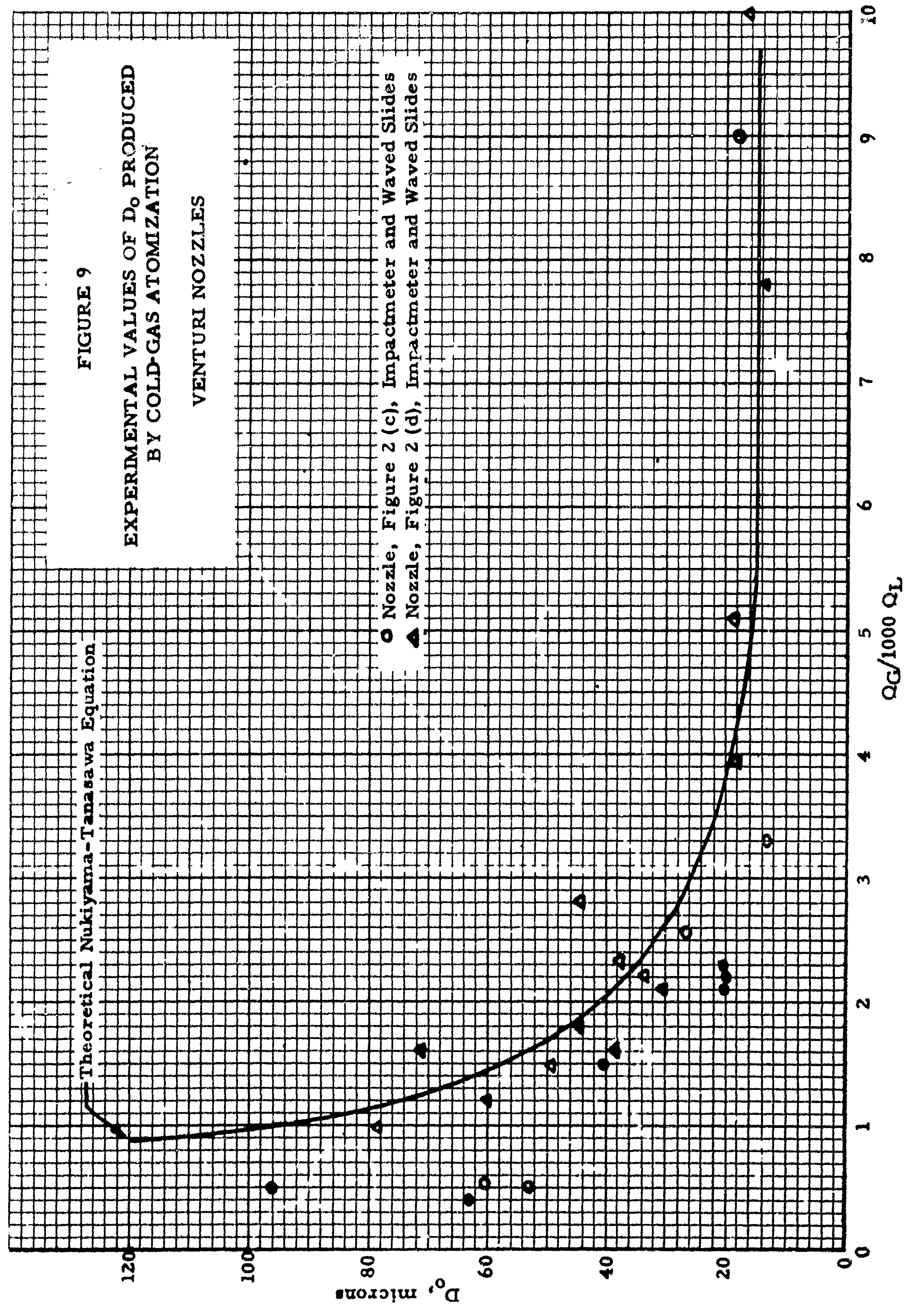


FIGURE 9

EXPERIMENTAL VALUES OF D_o PRODUCED BY COLD-GAS ATOMIZATION

VENTURI NOZZLES

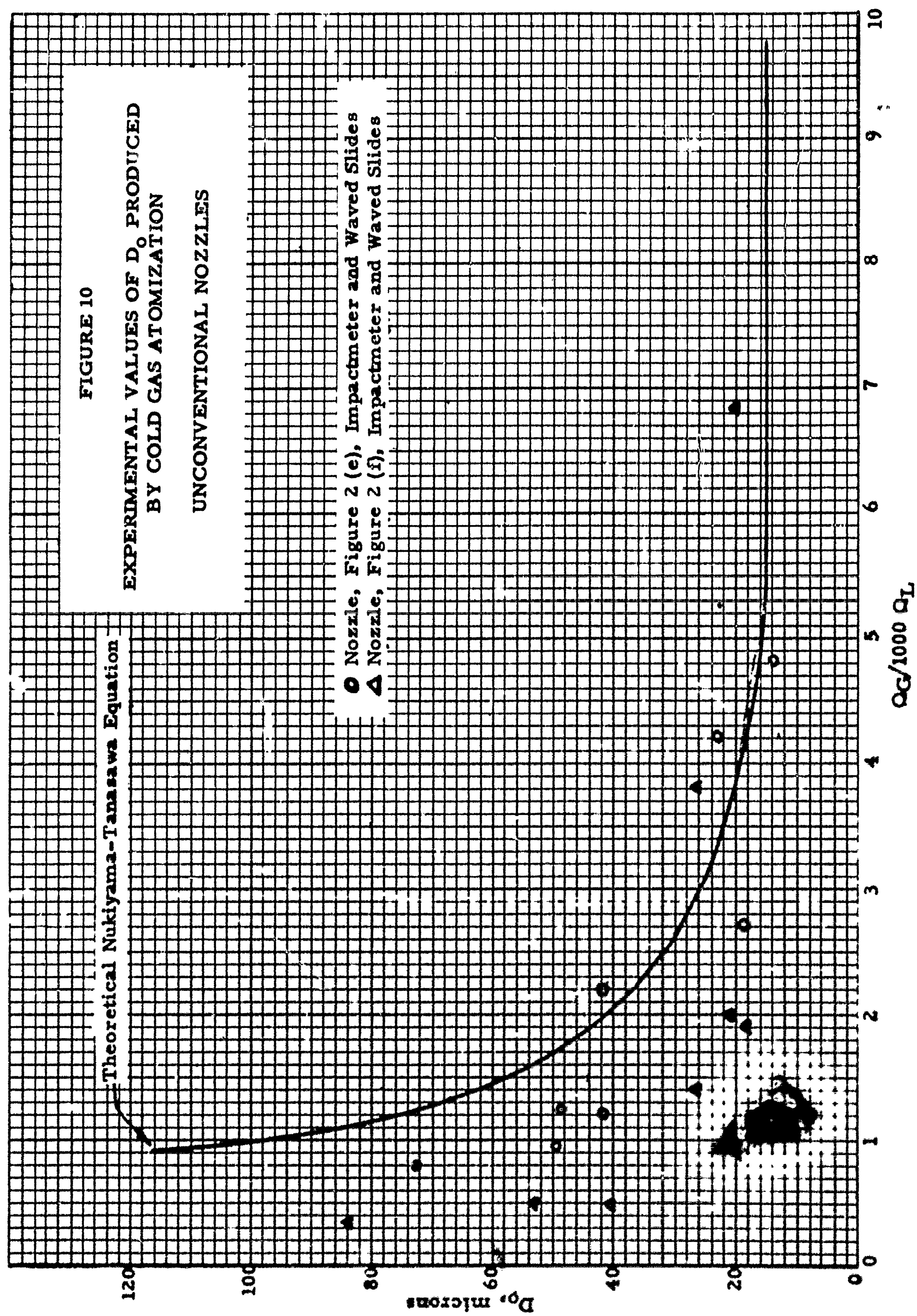


FIGURE 10
EXPERIMENTAL VALUES OF D_o PRODUCED BY COLD GAS ATOMIZATION
UNCONVENTIONAL NOZZLES
Theoretical Nukiyama-Tanasawa Equation
Nozzle, Figure 2 (e), Impactmeter and Waved Slides
Nozzle, Figure 2 (f), Impactmeter and Waved Slides
D_o, microns
0
20
40
60
80
100
120
Q_G/1000 Q_L
0
1
2
3
4
5
6
7
8
9
10

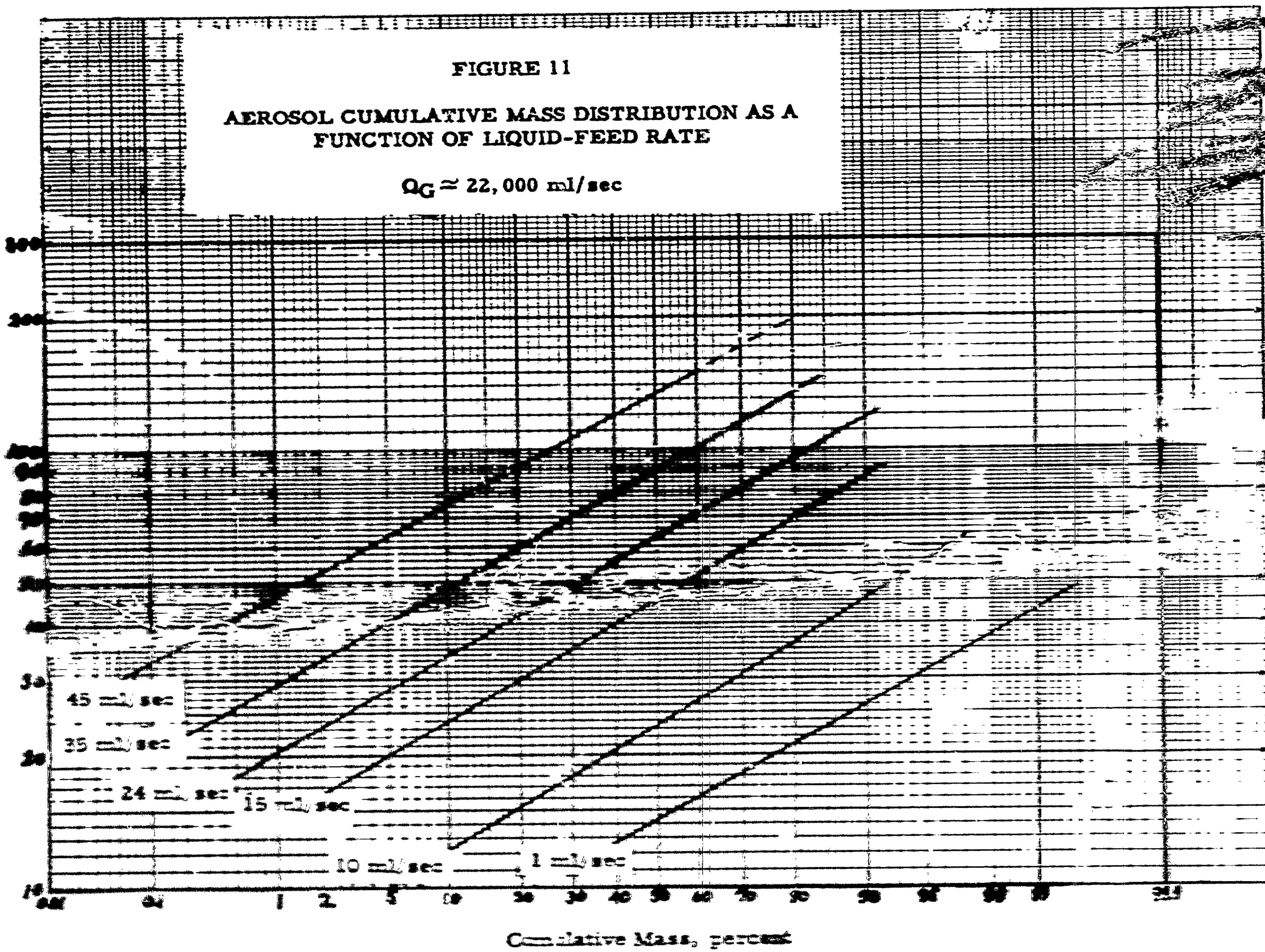


FIGURE 11

AEROSOL CUMULATIVE MASS DISTRIBUTION AS A FUNCTION OF LIQUID-FEED RATE

$Q_G \simeq 22,000$ ml/sec